February 2003 Editor: Regina R. Montgomery

SRM SPOTLIGHT

In this Issue…

New SRMs

Drugs of Abuse in Human Hair I and II SRM 2379 and SRM 2380

Drug abuse continues to be a major problem in the U.S. Considerable effort is expended to identify users of illegal substances to protect public health and to reduce demand for such substances. Urine drug testing is widely used to detect evidence of drug use. However, urine drug testing has a limited detection window, generally within 48 hours of when a drug was used. In recent years, much attention has focused on testing hair for drugs of abuse, because drugs can be detected in hair long after the drug use has stopped. Analysis of hair for drugs of abuse is being used to furnish data for medical evaluations, criminal and civil court proceedings, pre-employment personnel screenings and security clearance checks, random drug tests, and disciplinary actions. A number of controversial issues are related to hair testing, however, one of which is the reliability of measurements of drugs in hair. To address this need, NIST has developed two drugs of abuse in human hair Standard Reference Materials, SRM 2379 and SRM 2380 that can be used to check the accuracy of analytical methods used to determine drugs of abuse in hair.

SRMs 2379 and 2380 consist of human hair taken from one drug-free individual and spiked with the analytes of interest: cocaine (COC), benzoylecgonine (BZE), cocathylene (CE), amphetamine (AMP), methamphetamine (MAMP), and phencyclidine (PCP) in SRM 2379; and morphine (MOR), codeine (COD), 6-monoacetylmorphine (MAM), and tetrahydrocannabinol (THC) in SRM 2380. The concentrations of these substances were determined at NIST using two independent analytical techniques for a given analyte: one involving gas chromatography/mass spectrometry (GC/MS) and the other involving liquid chromatography/mass spectrometry (LC/MS). The certified values for these analytes are equally weighted means of the two methods, reported on an as-received basis in mass fraction units, with the associated uncertainties expressed at the approximate 95% level of confidence. The certification of both SRMs is valid until 1 January 2008, within the measurement uncertainties specified, as long as the SRMs are handled and stored in keeping with the instructions given in its certificate. NIST will monitor these SRMs over the period of their certification. If substantive changes occur that affect the certification before the certificates expire, NIST will notify the purchaser. Certified information values are listed in the NIST Certificate of Analysis for Standard Reference Material® 2379, issued 23 October 2002; and for SRM 2380, dated 29 October 2002.

One unit of SRM 2379 and SRM 2380 consists of one bottle, each of which contains approximately 100 mg of hair segments. The Certificates of Analysis lists the mass fractions for each analyte, expressed in ng/mg.

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New SRMs continued…

Standard Reference Material 2298 Sulfur in Gasoline (High Octane)

Federal, state, and foreign nations' guidelines and standards for the release of sulfur from high-octane gasoline to the atmosphere are constantly being tightened. Certain high-performance automobile engines, as well as aircraft engines require high-octane gasoline, but these engines also must reduce their sulfur emissions to the atmosphere, to comply with ever more stringent guidelines. To ensure that engine emissions meet these guidelines, the manufacture of high-octane gasoline with sharply reduced sulfur content is required. Manufacturers must show that the use of their products will ensure that engines meet emission guidelines.

NIST announces the release of reference values for its SRM 2298 Sulfur in Gasoline (High Octane), which will be applied to evaluating methods and calibrating instruments used to determine total sulfur in gasoline or materials of similar matrix. A unit of SRM 2298 consists of five amber ampoules, each one of which contains about 20 mL of gasoline, which was donated by Exxon Mobil Company (Fairfax, VA).

The certified value is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS). Homogeneity testing was done by X-ray fluorescence spectrometry. The uncertainty in the certified value, expressed as an expanded uncertainty, is calculated according to the method in the ISO Guide, and is based on a 95% prediction interval. The certification is valid until 31 December 2008 and is considered to be stable during the period of certification, but it is nullified if the SRM is damaged, contaminated, or otherwise modified.

Certified Value (mass fraction)
Sulfur: 4.7µg/g ± 1.3 µg/g

Standard Reference Material 2299 Sulfur in Gasoline (Reformulated)

Federal, state, and foreign nations' guidelines and standards for the release of sulfur from reformulated gasoline to the atmosphere are constantly being tightened. Gasoline had to be reformulated with certain oxygen-containing compounds because of the ban on tetraethyl lead as an anti-knock compound and also because of requirements for reformulation that would reduce automotive emissions of ozone/smog precursors. In addition, automotive emissions of sulfur to the atmosphere must be curtailed, to comply with ever more stringent guidelines. To ensure that engine emissions meet these guidelines, the manufacture of reformulated gasoline with sharply reduced sulfur content is required. Manufacturers must show that the use of their products will ensure that engines meet emission guidelines.

NIST announces the release of reference values for its SRM 2299 Sulfur in Gasoline (Reformulated), which will be applied to evaluating methods and calibrating instruments used to determine total sulfur in gasoline or materials of similar matrix. A unit of SRM 2299 consists of five amber ampoules, each one of which contains about 20 mL of gasoline, which was donated by Exxon Mobil Company (Fairfax, VA).

The certified value is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS). Homogeneity testing was done by X-ray fluorescence spectrometry. The uncertainty in the certified value, expressed as an expanded uncertainty, is calculated according to the method in the ISO Guide, and is based on a 95% prediction interval. The certification is valid until 31 December 2008 and is considered to be stable during the period of certification, but it is nullified if the SRM is damaged, contaminated, or otherwise modified.

Certified Value (mass fraction)
Sulfur: 13.6 µg/g ± 1.3 µg/g

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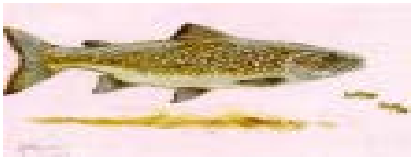
New SRMs continued…

SRM 1946 Lake Superior Fish Tissue

This SRM has been developed for evaluating analytical methods for determining polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, fatty acids, extractable fat, methylmercury, total mercury, and selected trace elements in fish tissue and similar matrices. Of particular interest are persistent environmental and tissue contaminants such as PCBs and chlorinated pesticides, some of which are suspected carcinogens or have been banned for other reasons; and mercury and methylmercury, which are highly toxic, the latter to nerve tissue. SRM 1946 is the first to provide certified values for three non-*ortho*-substituted PCBs that are considered the most toxic because of their structural resemblance to 2,3,7,8-tetrachlorodibenzo-*para*-dioxin (TCDD).



In addition, this SRM is intended for analysis of food proximates (fat, protein, and carbohydrates) and other constituents of interest in food analysis. For example, fish containing more than a level of methylmercury established by regulation is not to be consumed.

Certified values are provided for 30 PCB congeners and 15 chlorinated pesticides, respectively. These values were obtained via two or more independent analytical techniques, based principally on chromatographic and mass spectrometric methods. Certified values also are given for total extractable fat and 13 individual fatty acids; these are based on measurements made by NIST and collaborating laboratories. Certified values for methylmercury, total mercury, arsenic, and iron are based on results of two or more independent analytical procedures, performed at NIST and collaborating laboratories.

SRM 1946 also provides reference concentration values for 12 PCB congeners, 2 chlorinated pesticides, 12 fatty acids, certain proximates and elements, and caloric content. Certified, reference, and information values are listed in the NIST Certificate of Analysis for Standard Reference Material® 1946, issued 7 October 2002.

One unit of SRM 1946 consists of five bottles, each of which contains approximately 10 g to 12 g (wet basis) of frozen (not freeze-dried) tissue homogenate. This SRM is currently in preparation.

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SRM 2387 Peanut Butter

The Nutrition Labeling and Education Act of 1990 requires that information for selected nutrients is provided on labels for processed foods. In response, NIST has been working to provide food-matrix SRMs with values assigned for the required nutrients. SRM 2387 Peanut Butter is the most recent SRM in this series. SRM 2387 is intended for use as a primary control material for assigning values to in-house control materials and to validate methods for measuring nutrients such as fat, protein, calcium, iron, calories, and vitamins. This is the first food-matrix reference material available from NIST for which values are also assigned for individual amino acids and for aflatoxins.

To study the robustness of analytical methods, AOAC International developed a nine-sector triangle in which foods are positioned based on their fat, protein, and carbohydrate content. The idea was that one or two foods within each sector should be representative of other foods within that sector when validating an analytical method. Similarly, one or two food-matrix reference materials in each sector can be used as control materials for other foods within that sector. With the release of SRM 2387, one or more reference materials in each of the nine sectors are available from NIST. This SRM is currently in preparation.

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New SRMs continued…

Diquat Dibromide Monohydrate in Water SRM 3072

Standard Reference Material (SRM) 3072 is a solution of diquat dibromide monohydrate (Chemical Abstracts Registry Number 85-00-07) in water intended primarily for use in the calibration of chromatographic instrumentation used for the determination of diquat dibromide monohydrate. This SRM can also be used to fortify aqueous samples with known amounts of diquat dibromide monohydrate. A unit of SRM 3072 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Diquat Dibromide: The certified concentration value [1,2], given below, is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using liquid chromatography (LC). A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Diquat Dibromide Monohydrate 39.7 mg/kg ± 0.8 mg/kg

A unit of SRM 3072 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

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Inorganics in Marine Sediment SRM 2702

Standard Reference Material (SRM) 2702 is a marine sediment collected at the mouth of the Baltimore Harbor, Baltimore, MD. SRM 2702 is intended for use in evaluating analytical methods for the determination of selected elements in marine or fresh water sediment and similar matrices. All of the constituents in SRM 2702, for which certified, reference, and information values are provided, were naturally present in the sediment material before processing

Certified values for concentrations, expressed as mass fractions, for 25 elements. The certified values are based on the agreement of results from two or more chemically independent analytical techniques obtained at NIST and collaborating expert laboratories [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is very near the site where SRM 1941 and SRM 1941a were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 70 μm (100 % passing), homogenized in a cone blender, radiation sterilized at 33 kGy to 45 kGy (60Co) dose, and then packaged in screw-capped amber glass bottles each containing approximately 50

A unit of SRM 2702 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment material. This SRM is currently in preparation.

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SRM News

SRMs 3194, 3195, and 3196 will no longer be produced. These SRMs will be available until the current supply is exhausted. In place of these SRMs, NIST-traceable conductivity standards can be prepared to an accuracy of about 0.04 % using SRM 999 (KCl) and > 16 Mohm·cm water. Customers interested in preparing the primary standards will be able to obtain the IUPAC document and instructions for solution preparation and uncertainty calculations by contacting the technical division or from the SRM website.

We now have our SRM Special Publications 260's posted on our website for your convenience.

If you would like to receive your newsletter via e-mail, please send your e-mail address to:

Spotlight@nist.gov

NIST SRM Exhibit Schedule

American Academy for Forensic Science (AAFS**) -** February 17 –22, 2003, Chicago, Illinois

The Pittsburgh Conference (PITTCON 2003**) -** March 9-14, 2003, Orlando, Florida

American Chemical Society (ACS) - March 23-27, 2003, New Orleans, LA

American Welding Society (AWS) - April 8–10, 2003, Detroit, MI

NOBCCHE - April 14-19, Indianapolis, IN

ACHEMA – May 19-24, 2003, Frankfurt, GERMANY

Biotechnology International Convention & Exhibition (Bio 2003) - June 22-25, 2003, Washington, D.C.

American Association of Clinical Chemists (AACC) – July 20-24, 2003, Philadelphia, PA

American Chemical Society (ACS) - September 7-11, 2003, New York

Renewal SRMs

SRM 2391b PCR Based DNA Profiling

This Standard Reference Material (SRM) is intended primarily for use in the standardization of forensic and paternity quality assurance procedures for Polymerase Chain Reaction (PCR)-based genetic testing and for instructional law enforcement or non-clinical research purposes. This SRM can also be used for quality assurance when assigning values to in-house control materials. It is not intended for any human or animal clinical diagnostic use. Note that SRM 2391b is slightly modified from SRM 2391, in that there is more emphasis on Short Tandem Repeats (STRs) and less emphasis on D1S80 reflecting the growing interest and utility of STRs. Additional information on each STR locus can be found at a NIST-sponsored database on the internet: http://www.cstl.nist.gov/biotech/strbase .

This SRM is composed of well-characterized human deoxyribonucleic acid (DNA) in two forms: genomic DNA and DNA to be extracted from cells spotted onto filter paper. A unit of the SRM is composed of 12 frozen components packaged in one box.

Other Renewals Now Available

SRM 1473b Low Density Polyethylene Resin
SRM 3192 Aqueous Electrolytic Conductivity
SRM 2034 Holmium Oxide Solution

Revisions - - - - - - - - Certificate Revisions – Are you Using These Materials?

Below is a list of our most recent certificate revisions. To gain maximum benefit from a NIST SRM, the certificate in possession must be current. NIST updates certificates for a variety of reasons, such as the extension of the certificate date or to include additional information gained from stability testing. If you do not have the most recent certificate for your material, download a copy from the website at: www.nist.gov/srm , or contact SRM at: telephone (301) 975-6776; fax (301) 926-4751; or email: srminfo@nist.gov.

SRM 870 Infrared Transmission Wavelength
Chromatographic conditions updated.

SRM 909b Human Serum
Reference values for total bilirubin added, decertification of the analyte glucose.

SRM 955b Lead in Bovine Blood
New expiration date: This material is valid until 03 March 2009.

REVISIONS continued…

SRM 1674 Carbon Dioxide in Nitrogen 7% mol/mol
New expiration date: This material is valid until 25 March 2008.

SRM 1678c Carbon Monoxide in Nitrogen 50 umol/mol
New expiration date: This material is valid until 25 March 2008.

SRM 1681b Carbon Monoxide in Nitrogen 1000 umol/mol
New expiration date: This material is valid until 20 March 2008.

SRM 1687b Nitric Oxide in Nitrogen 1000 umol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 1921a Infrared Transmission Wavelength
New expiration date: This material is valid until 31 December 2008.

SRM 1980 Positive Electrophoretic Mobility
New expiration date: This material is valid until 01 September 2007.

SRM 2631a Nitric Oxide in Nitrogen 3000 umol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 2639a Carbon Monoxide in Nitrogen 1% mol/mol
New expiration date: This material is valid until 06 March 2008.

SRM 2640a Carbon Monoxide in Nitrogen 2% mol/mol
New expiration date: This material is valid until 11 March 2008.

SRM 2641a Carbon Monoxide in Nitrogen 4% mol/mol
New expiration date: This material is valid until 14 March 2008.

SRM 2735 Nitric Oxide in Nitrogen 800 umol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 2736a Nitric Oxide in Nitrogen 2000 umol/mol
New expiration date: This material is valid until 01 April 2008.

SRM 8506 Water in Transformer Oil
Coulometric reference values updated.

SRM 3195 Aqueous Electrolytic Conductivity (Lot # 011804) Expiration Date Has Been Extended
The expiration date for SRM 3195 Aqueous Electrolytic Conductivity (100,000 µS/cm) has been extended. This material is now certified until 24 May 2004. **(See note on SRM 3194, 3195, and 3196 in SRM News section in this newsletter).**